GENTRAL FILES NUMBER 44-10-257

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ChemRisk Document No. 3040

This document consists of___ pages and____ No. 3 of 6 copies. Series A.

L. B. Emlet

B. C. Nylen W. Q. Smith

J. P. Sinclair

Reading File

Central File

10-18-44 100 Area

Production

J. P. Sinclair

Mr. L. B. Emlet

Discharge of Metal

Please begin the discharge of metal for use in the 200 Area again on Saturday, October 21, 1944. Discharge should be at the rate of 3 buckets of 264 slugs each every other day from October 21 on until further notice.

Production Superintendent

JPS/maw

LASSIFICATION CANCELLED

FEB 12 DATE

For The Atomic Energy Commission

Chief, Declassification Branch

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CENTRAL FILES NUMBER

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Those Eligible Date_ To Read the Attached Subject_ J. P. Similaly L. D. Bellet Before reading this document, sign and date below Date Date Name

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Use MC User 9 15/95
Technical Information Officer Date

L. B. Emlet

B. C. Nylen

W. Q. Smith

J. P. Sinclair

Central File

Reading File

L. S. Emlet

J. P. Sinclair

Metal Discharge Schedule

Attached is a tabulation showing expected use of material now in the canal, and schedule for discharge of material which it is estimated will be processed by December 31. Variations required in discharge dates in order to prevent interference with necessary operation will be satisfactory within the limits of plus or minus three days, but the preferred policy should be to leave the metal in the pile and approach 27 day aging rather than to discharge it too far ahead of schedule.

The 302 slugs in Bucket No. 29 should be reduced to 264 by transfer of 38 slugs to Bucket No. 49, and Bucket No. 49 which will then contain 200 slugs can be brought up to 264 as additional slugs become available, and used in one of the half ton batches to replace a bucket scheduled for discharge.

Please note that Bucket No. 26 discharged 9/5 and the eight buckets discharged 9/8 are to be held as nominal 60 day material for runs 252 - 260. Please note also that no pile discharge is scheduled for the periods 9/9 to 9/20 inc., and 9/28 to 10/16 inc., and that only one bucket needs to be discharged on 9/21.

Production Superintendent

JPS/maw

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- ja			A CONTRACT			
200 Area Run			Number of 264	Charge to	Sept. 5 Bucket	Discharge from Pile
Nos.	Age-Days	Batch Size	Slug Buckets	Date	Number	Date
218, 219, 220 221, 222, 223 224, 225, 226 227, 228, 229 230, 231, 232 233, 234, 235 236, 237 238, 239 240, 241 242, 243 244, 245 248, 249 250, 251 254, 253, 254 255, 266 267, 268 263, 264 265, 266 267, 268 269, 270 271, 272 273, 274 275, 276 277, 278 279, 280 281, 282 283, 284 285, 286 287, 288 289, 290 291, 292 293, 294 295, 296 297, 298	35 35 60 59	1/3 Ton 1/2 Ton 1/3 Ton 1/3 Ton 1/3 Ton 1/2 Ton 1/2 Ton 1/2 Ton	33333333333333333333333333333333333333	9/12 9/14 9/16 9/28 9/30 10/13 10/15 10/17 10/19 10/23 10/25 10/27 10/30 11/1 11/3 11/16 11/28 11/26 11/28 11/26 11/28 11/26 12/2 12/4 12/6 12/12 12/14 12/16 12/12 12/14 12/16 12/12 12/16 12/12 12/12 12/14 12/20 12/22	4, 11, 1 50, 3, 54 32, 24, 19 64, 62, 6 53, 17, 60 67, 15, 44 10, 61, 12 29, 23, 41 43, 63, 56 69, 58, 51 47, 34 4 57, 19	8/9 8/9 & 10 8/10 & 14 7/29 & 8/1 3/1, 2 & 4 8/4 & 5 6/26 & 8/9 7/24, 7/27, 8/2 8/4, 9/21 9/23 9/25 9/8 10/17 10/19 10/21 10/23 10/25 10/27 10/29 10/31 11/2 11/4 11/6 11/8 11/10 11/12 11/14 11/16 11/18 11/10 11/12 11/14 11/16 11/18 11/10 11/12
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pages and of figures. No. of of 33 copies, Series A.

figures.

W. K. Chelie M. 1966 September 1, 1966 Miller

From: J.A. Swartout To: Section C-III

Ref.: Daily Plant Reports NCK from WLS

Comparison of Clinton vs. Hanford with Scavengers

			Extra	Extraction	lst By-Pro	lst By-Product	Pro	lst roduct	By-Product. BiPO,	Product BiPO,	LaF3 (Prod.) Cross-over	Prod.)	Owerall:	111: 70n	For E. L. Tony, Suppliers
á	Flow-	0, C	<u> </u>	گ ^و	3 00 0	≫ €	86 C	20 00 00 00 00 00 00 00 00 00 00 00 00 0	88 0	745	200 a	200	P6 50	76	S over one C
AUG 1	00015			3	202				2007	3	200		7	3	A COMMENT OF THE COMM
154~158	Clinton	πX ₁₁	1.5	0.72	0.3	1.3	1.9	1.2	0.5	(2)	۲.	7.7	5.8	4.4	HF in LaF3 prod. red. to 0.5M
159-161	-		1.3	0°2	9*0	1.6	1.1	0.8	0.8	(4)	۳, ۲	1.8	6.4	4.1	1)Sep. tank for each step. 2)H3PO4 in BiPO4 by-product red. to 02M
162-169	72- 44-	=	1,8	9.0	7.0	1.6	1.6	٦,٦	9.0	(3)	3.6	1.0	5.9	4.22	Ditto
170-177	Hanford	T. A.	1.7	0.75	3.7	1.65	2.0	۲. د	2.0	0	2.5	0.85	11.5	57.7	4.45 Ce-Zr Scav. { 1st strike 2nd Strike Ce-Zr
178-185			0°4	0.7	3,6	1.6	3.0	r- 	2,4	(3)	2.1	6.0	15.1	7.0	Ditto and: 1) H ₂ SO _L added to 40% UNH, 2) (178-182) 3 washes of 1st By-Product
136-193		~	2,9	0.8	3.7	7,6	2,3	۲°3	2nd B	2nd By-Prod. (Scav.)	2nd Prod. BiPO,	rod.	Overall- 2 Cycles	111-	
		Samuel Control of the San San							4°4	J.0	2,1	1.4	15.3 5.2	5,2	2 - Dec. cycles 1st: Ce-Zr, Ce-Zr. 2nd: Ce-Zr, Ce-Zr.
194-201			2.4	0.8	2.9	2,5	1,0	2.1	3.0	0.5	8.0	7.4	10.1	5.4	2 - Dec. cycles lst: Ce-Zr,Ce. 2nd: Ce-Zr,Ce.
202-209		=	2.0	1.0	3,3	1.2	0.7	ب ج	0.7	0.1	0.2	1.6	7.0	5.4	2 - Dec. Cycles Jst. Ce. 7r. Ce.
iffi. o See arren (c. Eniopse or plus or grandler	A Comment			And the state of t	macon Megion (10 an		ż								By-Prod. Prod. No scav. 0.05M(NH ₄) ₂ S

J. P. Sinclair

DEPARTMENT

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FROM W. Q. Smith

CF-44-9-10

DEPARTMENT

DATE DEC 6 196

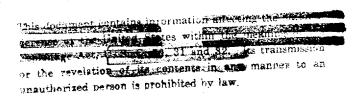
IN RE: Proposed 200 Area Operating Schedule

For The Atomic Energy Commission

Attached is a proposed operating schedule for the 200 Area for Runs 218 and 268 inclusive. Two primary objectives dictated this arrangement: (1) Production of the maximum quantity of product by the end of October, and (2) continuation of 204 Building operation using a proposed Hanford procedure and Hanford size batches.

The pertinent features of this schedule are:

- 1. Three 1/3 ton batches will be metathesized together in Room D giving a Hanford size batch in Room D and 204 Building and Hanford quantities of lanthanum in the product precipitation step of the crossover (at present 50% more lanthanum is used than is on the Hanford flowsheet). The demonstration runs are then more conveniently broken down into a group of nine rather than a group of eight runs.
- 2. The supernatent from the peroxide precipitation will be reacted with NaNO₂ to destroy the peroxide and returned to supply lanthanum for the first by-product precipitation. In the present series this is being returned to the product precipitation step with the result that a third lanthanum precipitation must be made and this worked up separately in order to recover the product which is carried by the lanthanum not removed in the centrifugation. B. A. Fries has tested a similar procedure except that he used KNO₂ to destroy the peroxide and then re-oxidized the product before returning it to the system. This re-oxidation is not considered necessary since a permanganate re-oxidation step is now being carried out immediately before the lanthanum by-product precipitation. J. B. Sutton has agreed to test the effect of returning the NaNO₂ treated peroxide supernatant at this point in the process, on by-product and product waste losses in the crossover.
- 3. In order not to hold up operations while waiting for the return of the peroxide supernatant it will be necessary to have two recycle batches in the process. This will involve about 2½ grams of product in each if the solubility is 50 mg per liter. Batches 208 209 gave a solubility of 59 mg per liter and it is anticipated that at least this low a solubility can be obtained on future batches.
- 4. Following the demonstration runs with CeZr-CeZr in each cycle and 60 day metal 16 runs will be made using ½ ton batches and one cycle operation (with by-product lanthanum fluoride) in order to catch up on the schedule delay caused by the continuation of the two cycle operation. This will delay the 60 day runs on the ammonium fluosilicate plus scavengers until metal which will be pushed immediately has cooled 60 days.



W. Q. Smith

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WQS/maw

200 AREA SCHEDULE

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Run	218, 221, 224,	23,	236, 242, 246, 246, 246, 246, 246, 250,	252, 255, 258, 251,	265,	9-7-44 WQS/maw

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CLINTON LABORATORIES

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Date 9/2/44 Those Eligible To Read the Subject (Classified Notebooks and Other Materials at Clinton Lab Attached to be made Available NEW) W. E. Kirst N. D. Whitaker Before reading this document, sign and date below Name Date Name R. L. DOAN

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Folder S-I & S-III M. F. Acken Semi-monthly reports of Separations Development Division Folder- Recommendations for plant procedures ✓ Problem Assignment Reports, SeI, S-II & S-III sections
✓ Folder - 205 Area Reports, W. Q. Smith to W. C. Kay BiPO, report, W. F. Underwood Recommendations for process conditions at Hanford - Report B. C. Nylen to W. C. Kay ✓ Memo - Plant dummy scavenger runs, 7-12-44, B. C. Nylen to W. C. Kay Clinton cell procedures (runs 186-195) Clinton concentration procedures and Room D solution make-up ✓ Semi-works handbook Semi-works standard procedures -Hanford flowsheet (9-1-44), J. B. Work / Notebook - CL-326 (R. H. Beaton notes) ✓Notebook - CL-479 (R. H. Beaton notes)

✓Folder - S-II semi monthly report file (R. H. Beaton copies)

✓Folder - Plant test correspondence (R. H. Beaton copies) Report - CL-1305, Clinton lecture notes (R. H. Beaton copy) Folders - adsorption test data (R. H. Beaton copies) Semi works extraction-decontamination procedure (R. H. Beaton copies)



Suggested by: N. P. ACKEN

Works.

Clinton Lab. Notebook #

> Sincerely yours, EXPLOSIVES DEPARTMENT - TNX

WETCOMET.

CHETCH LABORATORIES

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44-10-284

Date 10-21-44

Subject Summary of Runs 227 - 235

Through Isolation Copy # 6- S. S. English

By W. Q. Smith

To J. P. Sinclair

Before reading this document, sign and date below.

PMS 11/93/44

This document has been approved for release to the public by:

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October 21, 1944

27. Central File

To: Mr. J. P. Sinclair Department: Production

18. H. Worthington

From: W. Q. Smith Department: Production

In re: Summary of Runs 227 - 235 Through Isolation

9. M. D. Peterson

Run Number	1 - 235	227 - 235
% Yield	83.7	70.9*
% Waste	14.4	21.6
% Material Balance	98.1	92.5

*Corrected for loss in product content of recycle solutions on hand.

SUMMARY

This nine-run series was processed according to the same procedure used on the previous series (Hanford procedure with recycling of isolation supernatants) with the exception that Ce-Zr; Ce-Zr scavengers were used in both decontamination cycles with no (NH₄)₂SiF₆. With the completion of these runs, sufficient plant-scale data have been obtained on the performance of scavengers and the complexing agent (NH₄)₂SiF₆, to lead to the conclusion that using (NH₄)₂SiF₆ with no scavengers in the decontamination cycles will reduce waste losses by a factor of five. Furthermore from plant data it appears that sufficient decontamination for Hanford level of operation can be obtained under these conditions. Comparison of the combinations tested in the plant is given in the discussion with data to support the above conclusions.

The flowsheet amount of NaNO₂ (0.1M) was used for pre-extraction treatment in this series. Extraction waste losses averaged 1.9%, slightly lower than results obtained previously with this concentration.

Decomposition of $\rm H_2O_2$ in isolation recycles with NaNO₂ has given no trouble. The amount of NaNO₂ necessary has increased to about 80% of the stoichiometric amount and the temperature rise has been $10-12^{\circ}$ C. over an addition period of about one hour.

The first peroxide precipitation solubilities in the series were 119, 83 and 89 mg per liter. Table on page 3 shows the variables which may affect these solubilities. The final peroxide precipitation solubilities were 16 and 53 mg per liter. For the purpose of this and subsequent reports the solubility is obtained by dividing the product content of the decanted supernatant plus washes by the neglection of the decanted

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October 21, 1944

To: Mr. J. P. Sinclair

Department: Production

From: W. Q. Smith

Department: Production

In re: Summary of Runs 227 - 235 Through Isolation

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For The Atomic Energy Commission

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Chief, Declassification Branch

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Through 2nd by prode ppt. 41R	101.3	101.3 1.02.8 1.04.3 1.03.2 98.6 1.06.7 98.9 1.07.0 96.4	104.3	103.2	986	106.7	98.9	107.0	96.4		
Through decont. cycles 44P	101.4	100.0	102.7	93.5	114.1	98.7	95.0	98.7	117.5	90.7	
Through final peroxide ppt. IFP**	92.5					•	MINISTER OF	-		***************************************	
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#151R and DIP for runs containing recycled product are based on the average value of the 11M plus the amount of recycled product added.

***Corrected for sain in product content of recycle solutions on hand and based on total 11M.



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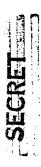
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Factors Affecting Peroxide Solubility

AND AND ASSOCIATED TO THE CONTRACT OF THE PARTY ASSOCIATION AND ASSOCIATED AND ASSOCIATION	The state of the s					
Run Numbers	Fe (14)	Zr (M)	Acidity	H ₂ O ₂ (%)	Settling	Solubility**
			(N)		Time	mg/liter
227-228-229 0.0067	0.0067	0.00006	0.8	5°6	9 hrs.	119
230-231-232	0°0065	N11	0°2	8.6	20 hrs.	83
233-234-235	0°0063	Nil	0,8	8.3	15 hrs	88
100 min 100 mi						

*BP-L, 153WS, D3GW and D3WW for runs contianing recycled product are based on the average value of the like plus the amount of recycled product added.

**Value after decenting and washing.



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With the completion of this series of runs the final demonstration of the recommended Hanford process has been made. Results confirm that the recommended process will give adequate decontamination. Waste losses, however, although they may not be considered excessive, have averaged five times as great with the Ce-Zr; Ce-Zr scavenger as with ammonium fluosilicate (no scavenger) in the cycles. It has been predicted that at Hanford concentrations the losses will be somewhat less but there seems to be no reason for doubting that they could not be lowered by the same factor of five by omitting scavengers. At Clinton the yield loss for two cycles is 9.4% greater with Ce-Zr; Ce-Zr, than with (NH,),SiF, alone in both cycles. (Waste losses were 5.9% per cycle with Ce-Zr; Ce-Zr scavengers, average of 64 results, and 1.2% per cycle with (NH4)2SiF6, average of 16 results. See table below.)

Run Nos.	Seavengers First Cycle	Scavengers Second Grale	dF Thru 2 Cycles 45P	dF Thru Cross-Over Dl-S	To Waste 1st Cycle	% Waste 2nd Cycla
194-201	Ce-Zr; Ce	Ca-Zr; Ca	5.3	6.7	3.9	3.8
202-209	Ce-Zr; Ce	(NH ₄) ₂ SiF ₅	5 e 3	7.1	4.0	و.
218-226	$\binom{\text{Ce-Zr; Ce-Zr}}{\binom{\text{NH}_{\downarrow}}{2}\text{SiF}_{6}}$	(NH _{L,}) ₂ sif ₅	5.7	7 c ls	4.5	1.5
186-193	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.6	6.3	ંઠ₀0	6.5
210-217	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.8	7 ₀0	6 .9	4.7
227235	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.8	7:4	5.7	5.Ò

No Clinton plant runs have been made with fission products, and with ammonium fluosilicate but without scavengers in both of the cycles. However, based on the results obtained to date it appears that the omission of scavengers but inclusion of ammonium fluosilicate will give decontamination factors of greater than 105 before the cross-over and greater than 107 after the cross-over. Thus the comparison of runs with Ce-Zr; Ce in the 2nd cycle vs those with (NH_L)₂SiF₆ alone in the 2nd cycle (Ce-Zr; Ce in the first cycle of both series) favors the complexing agent. (dF was 5.3 on both at end of 2nd cycle, 7.1 with (NH_L)₂SiF₆ vs. 6.7 with Ce-Zr; Ce at end of the cross-over.) A similar comparison with Ce-Zr; Ce-Zr scavengers but in this case with (NHL) SiF; also in the first cycle when omitting the scavenger from the second, does not give as clear-cut a case, but still favors the complexing agent. (dF's were 5.7 and 7.4 with (NH₄)₂SiF₅ in 2 cycles, Ce-Zr; Ce-Zr in one vs. 5.74 and 7.2 with Cs-Zr; Ce-Zr in both cycles.) Since the improvement does not show up until after the cross-over it appears that ammonium fluosilicate is more effective in removing specific activities which are not readily removed in the cross-over than are the scavengers. Unfortunately this point cannot be checked by analyses for specific fission products since the activity after the crossover is too low. Since all laboratory comparisons of scavengers vs. ammonium fluosilicate were only rerried through the two cycles and since laboratory centrifuging tends to increase the effectiveness of scavengers, but not of ammonium fluosilicate, it is not inconsistanidothatulaboraturpym reaultestimane mational defense of the United States within the meaning of the the scaveagers

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In comparing the decontamination results the effect of cooling time has not been considered. This is permissible since the elements which control decontamination, Cb, Zr and La, make up the same fraction of total gamma activity at 60 days (77%)* as at 30 days (73%).*

Actual measurements have been made on radiation during the product isolation steps and during the cross-over steps to check on the decontamination required at Hanford when processing material at full product levels. A factor of 107 at the end of the cross-over seems to be adequate, but not overly conservative. At the end of the two decontamination cycles, the greatest radiation is obtained in the vicinity of the 10N HNO3 solution. Readings in the vicinity of this tank corrected to the case where the operator is at a distance of ten feet and is protected by 1 foot of concrete indicate that a factor of only 1.5 x 104 is required to reduce radiation to .01 mr/8 hours. Under these conditions an operator could spend 5 minutes within 1 foot of the tank when it contains a charge without exceeding his daily dose. This has a bearing on the choice of decontamination procedure since it indicates that the factor of 107 at the end of the cross-over should be controlling and 105 at the end of the 2 cycles is not a justifiable limit.

Summarizing, it is certain that waste losses can be decreased appreciably by emitting scavengers and it appears very likely that adequate decontamination (greater than 107 after the cross-over) can be obtained with ammonium fluosilicate alone.

W. Q. Smith

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1. M. D. Whitaker

2. S. W. Pratt

2. S. W. Pratt

3. J. P. Sinclair

No. L. of 3 copies. Series A

CLINTON LABORATORIES

DATE September 8, 1944

To M. D. Whitaker

DEPARTMENT

CLINTON LABORATORIES

FROM J. P. Sinclair

IN RE: Revised Production Schedule

DEPARTMENT

CENTRAL FILES NUMBER

14-9-157

Attached is a revised schedule of production for the 200 Area based on memo from W. Q. Smith to J. P. Sinclair of 9-7-44 on Proposed 200 Area Operating Schedule. The latter was discussed and recommended for your approval in a meeting of the Steering Committee yesterday.

You will note that the schedule includes only material to be finally isolated ready for delivery by December 31, and that the discharge schedule for material from the pile therefore ends on November 22. By the revised schedule 28 tons will be discharged from the pile in the period September 5 to November 22, instead of 32 tons in the period September 27 to November 30 shown in the schedule of August 25.

Assumptions made in preparation of the revised schedule are the same as those used in the schedule of August 25, except that 250° maximum metal temperature until November 22 has been definitely planned on, and allowance for decrease to 57,000 KWH/day from 62,000 KWH/day has been included for a fifteen day period during the reinstallation of the large No. 2 fan.

In order to follow the process outlined in Smith's memo, an expenditure of perhaps \$3,000 may be required for equipment changes in 204 Building. A request for a project is now being drawn up, and emergency approval will be requested. This contingency was not forseen because the recommendation for the proposed process arose from the experimental work now in progress with Runs 202 - 217.

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ASSIFICATION CANCELLED

DATE 950 6 1980

For The Alemie Library Commission

Production Superintendent

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			0.00 0.00 0.00	4.6		8/9 8/9&10 8/10&14	9/30 10/2 10/4	9/26 9/28 9/30	9/12 9/14 9/16	Sif ₆	CeZr, CeZr, SiF6	1/3 Ton (332	218220 221223 224226	2222
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W.C.K. Est. of 6/13/4	J.P.S. W. Est. Es	Total Product Delvr'd for Month	Product Deltr'd 80% Yield Basis	Product Disch'gd from File	Pile Zone	Discharge from Pile Date	Charge Ready to Ship	Charge to Room D	First Charge to Cell 1 Date	s Second Cycle	Process First Cycle	Batch Size	Age Days	Run Nos.	Ru

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CLINTON LABORATORIES

#3040

P. O. BOX 1991

1 - E. J. Murphy

2 - A. H. Compton KNOXVILLE 11, TENNESSEE

3 - M. D. Whitaker

September 2, 1944 44-9-54

The District Engineer U. S. Engineer Office Manhattan District P. O. Box E Oak Ridge, Tenn.

Attention: Major E. J. Murphy

Dear Sir:

The data which you have requested for your monthly report for August 1944 are given below:

(a)	Tons of slugs placed in production pile during August	9-57
(b)	Tons of slugs removed from production pile during August	9.59
(o)	Anticipated product delivery during September	22 g
(d)	Number of batches processed through Bldg. 205 during August	16
(e)	Number of research problems completed during August	8
(f)	Total number of Clinton Laboratories employees as of August 31, 1944	<u> 1</u> لبلر

Very truly yours,

M. D. WHITAKER, DIRECTOR CLINTON LABORATORIES

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itta File Those Eligible Date To Read the Attached Subject_ 9/29/44 Revised Metal Discharge Schedule Ву____ To Sinelair Before reading this document, sign and date below Date . This document consists of ... pages and ____ No. 3 of 6 copies, Series A.

B. C. Nylen W. Q. Smith

J. P. Sinclair Reading File

Central File

9-29-44 Production

J. P. Sinclair

L. B. Emlet

Production

Revised Metal Discharge Schedule Ref: Memo J. F. Sinclair to L. B. Emlet - 9/9/44

Confirming our conversation this morning please arrange to discharge from the pile during the next few days an additional nine buckets of 264 slugs each. This discharge is required to prevent possibility of there being no preperly aged metal available during the last week of October in case of improvement of the schedule given in the memo of 9/9/44. The situation will be reviewed again about October 15 with a decision to be made at that time on whether discharge will start again on October 17 as shown in the memo of 9/9/44 or whether the beginning of discharge will be delayed beyond October 17 because of improbable use of the subject nine buckets during the last week of October.

Please take advantage of this nine bucket discharge to abandon thermocouples no longer of great use at the outer edges of the pile, and to use the thermocouplesplugs so obtained to increase the number of thermocouples in the hot ring outside the poison in order to obtain more information on temperatures in this region.

Production Superintendent

JPS/maw

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This document has been approved for release to the public by:

CENTRAL FILES NUMBER 44-10-236

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#3040

Date 10-17-44 File 11/2	= 3
Subject Summary of Runs 218 -	8. G. English
226 Through Isolation	V. Cuglish
By W. Q. Smith	
To J. P. Sinclair	
	FILE CORN
Before reading this document, sign and data below.	
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This document has been approved for release to the public by:

Technical Information Officer Date

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October 17, 1944

To: Mr. J. P. Sinclair

Department: Production

From: W. Q. Smith

Department: Production

In re: Summary of Runs 218 - 226 Through Isolation

Run Number	1 - 226	218 - 226
% Yield	85.0	87.6*
% Product to waste	13.7	13.8
% Material balance	<i>9</i> 8.7	101.4

*Corrected for gain in product content of recycle solutions on hand.

SUMMARY

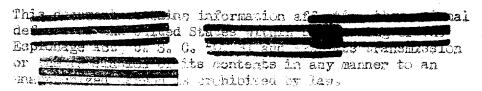
Demonstration of the Hanford process was continued with this series of runs, using the Hanford quantity of lanthanum in the cross-over product precipitation (two thirds of the amount previously used at Clinton) and combining 3 runs for KOH-K2CO3 metathesis, thus giving compositions and volumes of solutions from the metathesis step through isolation which were the same as those for Hanford. The supernatants from the peroxide first precipitations were recycled into the cross-over cycle at the by-product precipitation step after decomposition of the peroxide with sodium nitrite. Waste losses in the LaF3 by-product and product precipitation steps on the runs containing recycled product averaged about 1% higher than normal. Details of the recycling procedure are given in the discussion.

Extraction waste studies were continued using various NaNO2 toncentrations. From the results obtained, it appears that lower concentrations than are now used may be desirable.

The scavenger combination on this series was Ce-Zr; Ce-Zr, $(NH_L)_2SiF_6$ in the first cycle and $(NH_L)_2SiF_5$ alone in the second cycle. Excellent decontamination was obtained (dF = 7.4 through the cross-over) while waste losses (decontamination cycles only) were 6.0%. Comparison with other combinations is given in the discussion and tables.

Slightly higher peroxide solubilities were encountered on these runs than on the first runs with this procedure with no apparent explanation having been found as yet.





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October 17, 1944

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TH. R. Canall
Chief, Declassification Branch

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92,1 88.0 97.0 94.8	0.88	97,0	8.46	89.9	38	98.9		84,6	8,06
97.1 66.4 121.7 99.5	36.4	121.7	25.66	86:43	3,0	30%	80.9	102.5	34.47
92.9 98.4 101.0 70.1	98.4	101.0	70.1	01.6	99.7	5.72		್ಯ 88	88 88
95.3 101.9 98.2 106.0	201,9	98°2	06.0 <mark>1</mark> 3	02.1	(1,80)	6.66		82,0	33.3
153,2 100 100 100 100	28	38	- 8	8	76.2	176.2		77° 69%	263.4
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Run Number	218-226	218	21.9	220	221	222	223	224	225	226
Through lat hy-prod. ppt. 51R	105.8	101,3	101.3 108.3 121.2 101.6 109.6 112.1	121,2	101.6	109.60	112.1		7.80 8.60	105.6
Through and by prod pot 41R	10%,0	92,3	92,3 131,1 108,8 91,2 113,8 113,6	108.8	91.2	113.8	113.6	ကို	35.8 109.5	102.8
Through decont, cycles 44P	100.5	103.8	111.9	80.5	106,5	110.1	105.4	91.8	7.56	
Through final peroxide ppt. 1FPa*	101.4							7		

Individual Run Summaries

Run Numbers 218			•					_	
	18 219		220	221	222	223	221	22.5	226
Date Started 9-14		-	3-15	9-16	9-17	9-18	919	9-19	9-50
Age metal in days		38	******	38	39	38	୍ଦ୍ୟ	36.	37
gram UNH x 10 ⁶				2.59	1.83	1.73	2.30	1,80	1,56

*151R and DIP for runs containing recycled product are based on the average value of the 11M plus the amount ***Corrected for gain in product content of recycle solutions on hand and based on total 11M. of recycled product added.

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*	7	The state of the s		Valency and Address of the Party of the Part	
Run No.	186~193	194-201	202~209	210-219	210-219
Scavengers let ovele	Caron Contraction Carons	7 . 2 . 20	() a 20 - 00		
	77 00 6 77		00 (17)00	17-90 (.17-90	Ce-Zr; Ce-Zr, (MH,)SIF
and cycle	Ce-Zr; Ce-Zr Ce-Zr; Ce		(MH,) SIF	Ca-Zre Ca-Zr	MY S' EN)
ist by-product cake 71_BP-1			0 %) c	O True
			ついつ	- 0	
(150 prod. ppr. effluent 53ms	r.,	C	22	C 10	` ***
		2	· · ·	**・1	→ 3
can cylipton cake /1-br-2	7:7	7,0	ر د د	0 (*	C
		<i>y</i> .	3	100	↑
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Bun No.	218-226	2.8	210	200	100	223	203	221.	206	300
Extr. ppt. offluent 23MS		7,7) 	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	· (-)	2,6	10	, C	1.7	, C
let by-product cake 71-BP-1	ي ئارى	1.4	4.2	19	0	207	7.7		9) e
lst prod, ppt, effluent 53WS		7,7	1.9	o N	0	2,0	C	7.0	0	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \
2nd by prod. cake 71 BP-2		7,0	53	0,	0	0	0	2	0	, -
2nd prod. ppt. effluent 43WS		ا م	7.00	C	C	0	O N	, -, c	7.0	
3rd by-product cake 71-8P-3		70.0	700	7,0	7.0	0,5	7,0	0,3	0	0
Lafy by-product cake 71-BP-4*		2,5	1,24	1,6		2.2	2.5	10	, -) (4
Laf, product pot. effluent 153MS*		0,7	0	0,5	7,0	9,0		8	,	, (
1st metathests liquor D3CW*			2,0		-	0.2			10	
	ر د م		0,7			ر د د			7	-
Total 205 Building (Basis 114)	13.8			*****					÷ :	Ti
				-						-

*BP~4, 153WS, D3CW and D3WW for runs containing recycled product are based on the average value of the



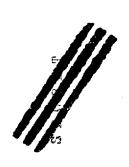
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Decontamination with Scavengers Tested

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Run Number	186~193	194-201	202~209	210=217	300 810	
					つなないでする	
Scavengers 18t cycle	Ce-Zr; Ce-Zr	Ce-Zr; Ce	Ce-Zr; Ce	e-Zr: Ce-Z	Ce-Zr: Ce-Zr. (NH.) SIF.	
2nd cycle	Ce-Zr: Ce-Zr	Ce-Zr: Ce	(NH,) SIF,	6-7r: Ce-7	OH)	
Through extr. ppt. 24P	8 0	000	0 0		2 T 2 (7) (1)	
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Through 13th by pro part and and the	200	(,	2,5		. 0.0	
Through 1st prod, ppt, 54P		, v,	4		3 1	
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ting and the state property of the state	ָ סֶ	ک ^ر م	က်		ئ ئ ئ	
Through 2nd prod ppt. (composite) 45P	5,6	(L)	ري ري			
Through Laft prod. slurry Dl-S	6.3	2	, , ,			
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DISCUSSION



Extraction Waste Studies

Pre-extraction treatment studies were continued varying the NaNO2 as follows: on the first three runs, 40% of the flowsheet amount (0.04M); on the next three runs, 100% excess added to the precipitator just prior to the strike (0.2M total); and on the last three runs, 25% of the NaNO2 (0.025M). Extraction losses for these tests averaged 1.5, 2.0 and 1.1% respectively. An average of 2.2% was obtained on the four runs of the previous series using double the amount of NaNO2 (0.2M) added in the dissolver. On 16 representative runs (194 - 209) using 0.1M NaNO2 the losses averaged 2.2, therefore, on the basis of these few runs, it is possible that lower concentrations might be desirable.

Scavengers

Decontamination on this series was assisted by the following combination of scavengers and complexing agent:

First cycle by-product pptn. - Ce-Zr; Ce-Zr product pptn. - (NH₄)₂SiF₆

Second cycle by-product pptn. - no scavengers product pptn. - (NH_L)₂SiF₆

Decontamination through the LaWa product slurry (DL-S) was the best obtained thus far and averaged 7.4 (dF) as compared to about 7.0 for previous combinations, while the dF at the end of the two cycles was 5.7 - well above the goal of 5.0. The waste losses were nearly as low as the best of the previous combinations: 6.0 for this combination against 4.9 for the combination Ce-Zr; Ce 1st cycle, (NH_L)₂SiF₅ 2nd cycle (extraction losses excluded). The results that have been obtained with scavengers and complexing agent indicate waste losses would be much lower and possibly still obtain sufficient decontamination for Hanford operations by using (NH_L)₂SiF₅ in both cycles with no scavengers.

Recycling of Peroxide Supernatants

Since the peroxide supernatants from the isolation step were introduced in the LaF₃ by-product precipitation step of the cross-over cycle, it was necessary to decompose the $\rm H_2O_2$ contained in these solutions before addition to the process. This was done in Room D as follows:

To 100 volumes of supernatant in tank D-8 cooled as low as possible (about 190 C.) add 15 volumes of 60% HNO2 (to make at least 1N) and then 17% NaNO2 solution at a rate such that the temperature does not exceed 300 C. When about 65% of the NaNO2 necessary to decompose the peroxide (peroxide content previously determined by analysis) has been added, remove a sample to ascertain if the peroxide has been all decomposed. If not, add more nitrite and resample. After the end-point has been passed, add a small amount of KMnO4 solution until a permanent pink color appears.

The reaction has been quite smooth with no unusual evolution of gas and has given about 9° C, temperature rise over the 1 hour and 20 minute $NaNO_2$ addition period. Only 60 - 65% of the stoichiometric amount of $NaNO_2$ has been required for the completion of the reaction.

After the above treatment, the supernatant was divided into two portions and used to furnish the lanthanum for the first shot of the by-product precipitations of the last two runs of the three-run batches. The first recycling in this series was done on the second three-run batch instead of the first so that two recycle solutions would be available, thus minimizing the possibility of process delays. The recycle was added before the KMnO, reoxidation and consequently the first lanthanum by-product precipitate was co-formed instead of preformed. By-product waste losses on runs containing recycled product were about 1% higher than normal while those from the product precipitation were variable so that no definite conclusions can be drawn on this step. The average was also about 1% higher than that for runs with no recycled product. Some of the product effluent waste samples of the next series of runs have been centrifuged in the laboratory with almost complete removal of the product. This definitely indicates that losses were due to inefficient centrifugation rather than incomplete reduction or carrying of the product. The table below shows the results of these tests.

Laboratory Centrifugation of 153WS Samples

Run No.	227	228	229	231	232
As received	2.3	0.4	1.3	1.1	2.7
After laboratory Centrifugation	0.0001	0.07	0.02	0.3	0.07

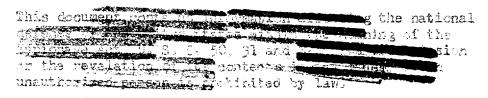
Reworking of Metathesis Caustic Wastes

On the basis of experience gained in the reworking of KOH- K_2CO_2 metathesis wastes in the two previous series, the following procedure was incorporated:

- 1. Centrifuge in regular manner after KOH-K2CO3 treatment.
- 2. Add 25 lbs. of 1% La solution and centrifuge again.
- Recentrifuge, if necessary.

Results were fairly good after the additional precipitation and centrifugation (1.3%). However, since the time was available, the effluent on each run was recentrifuged which decreased these waste losses to an average of 0.5%.







Isolation

High peroxide solubilities were encountered on these runs with no apparent explanation; however, variations in the iron, zirconium, acid and peroxide concentrations are being considered. Solubilities, along with these variables are shown in the table below. The solution for the final H2O2 precipitation was divided into two portions and each run separately under the same conditions. Solubilities after decantation and washing were 53 mg/liter and 20 mg/liter. Probably the washing technique was the cause of the different results obtained.

Run Nos.	Fe (M)	Zr (M)	Acidity (N)	H ₂ O ₂ (%)	Solubility mg/liter
218-219-220	0.008	0,00040	0.72	Service and	No pptn.
221-222-223	0.005	0.00006	0.83	9.4	(133 by fortifying) 83
224-225-226	0.005	0.00004	0,80	9.1	6 7

M. Q. Smith

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September 11, 1944

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SULMARY OF HUNS, 210 22 217 THROUGH

This series of runs will complete the special isolation test started with the previous series. Results presented here are through the decontamination cycles for this series. After completion of the isolation test, final results for both series will be reported.

Cerium-zirconium, cerium-zirconium scavenaers were used for decontamination in both cycles. Results were essentially the same as those obtained from the 60-day metal runs 186 - 193 which were processed with the same scavengers.

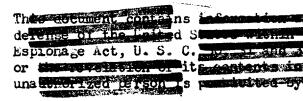
A number of changes in the extraction procedure are being tried in order to . improve waste losses in this cycle. On the first four runs of this series, a two hour direct strike using 90% of the oismuth solution followed by a thirty minute indirect strike with the remaining dismuth solution was used. On the last four runs, the sodium nitrite for pre-extraction treatment was doubled. aste losses in neither case showed a significant change. In the next series, 40% of the sodium nitrite will be used on four runs and then on the last four, 100% excess nitrite will be added in the precipitation immediately prior to the strike.

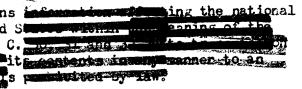
a table comparing the three decontamination procedures that have been used is given on Page 3 and an iron belance from crossover through solution of the crossover precipitates is liven on Fage 4.

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Jamus counts per gram UNH X 10°	Geta counts per gram UNH X 10°,	nge of metal in days	Date started	hun numoers
1.58	4.47	39	8-28	210
1.70	5.01	40	8-29	211
1.37	ں کوئ	40	8-29	212
1.82	5,78	42	8-31	213
1.63	4.63	42	8-31	214
1,52	4.47	43	9-1	215
1.62	5.95	41	9-3	216
1,39	68,4	41	9-3	217

The designated designation of the second of				Yi	Yield					
hun numbers	210-217	210	211	212	213	214	215	216	217	
6 Orio, prod.in extr.ppt.(24P)	96.2	98.7 87.7		102.7	100.0	102.7 100.0 105.6 102.7 92.9 96.4	102.7	92.9	96.4	····
o Oria, prod. after 1st by-pr.ppt. (51R)	108.5	108.8 96.4		10.0	113.0	11U.4	114.4	101.8	109.0	-
6 Origoproa in 1st proppt (54P)	93.6	9:0°4	88.8	3.TOT	90.6	70°3	79.6	95.9	95.5	
o Origoprodeafter 2nd oy-proppt. (41h)	96,0	90.9 87.6	£7.6	101.8	4.56	>9°7	0° 44 و 0	97.0	105.7	\$1
o Origoprodoin and proppt. (44P)	ÿ1.7	70.9 84.2	84.2	91.C	56.7	74.0	90.0	89,1	95.9	ų

			1	Reasured Losses	d Loss	es				
hun numoers	210-217	210	211	212	213	712	215		217	
Extrappt effluent (23kS)	1,9	1.8	2.0	<u>ر</u> د	1.7	1.4	2.4	بر ن	<i>3</i> °8	
lst Jy-prod.cake (71-8P-1)	ن ئ ، 7	2.4	2.7	3.4	٥، ب	ن 7،7	÷.9		4.5	
Lst From ppt effluent (53.8)	ω N	4.7	0° پ	ど。よ	ن ج 8	2°8	ွှ ဝ		2,1	
Zno Jy-prod.cake (71-8F-2)	ر ا	4.2	4.4	ယ ဇီ	<u>ئ</u>	٠. <u>٧</u>	3.4		3.4	
2nd Frod. ppt.effluent (43%S)	3.0	0.9		<u>ו</u>	0.6	ပ _္ 7	0.8		0.5	
l'otal masues thru decont.cycles	13.5	١٠٠١ م. ١٠٠١	1,41	12.0	14.4	11.9	14.5		14.4	
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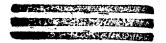
217	117.3	
216	107.0	
215	121.7)
214	115.5	
213	107.2	i ; ;
212	13.6	
211	101.1	· · · · · · · · · · · · · · · · · · ·
210	114.0 101.1 1 107.6 100.6 1	?
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hun number	210-217	210	211	212	213	717	₩.	્તાં	217	
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	7.7	4.1	7.7	5.8		707				
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t.,(45P)	ر ئ 8									
				-						

Comparison of Decontamination Processes

hun numbers	166-193	194-201	186-193 194-201 202-209 210-217 Ce=Zr,Ce=Zr,Ce=Oth cy. Ce=Zr,Ce=Isti(HH ₄) ₂ SiF ₆ 2nd Ce=Zr,Ce=Δr both cy 12.4 5.6 5.6 5.8	210-217
Process used	Ce-zr, Cs-Zrboth cy.	Ce-Zr, Ce both cy.		Ce-Zr, Ce-Zr both cy
Total wastes (Extractowastes excluded)	12.4	7.6		11.6
Decontamination (G df thru 45P)	5.6	5.3		5.8
	This docudence of the contraction of the contractio	This document contains in a defense of the UNITED Starts of the UNITED Starts or the trevelation of the pronunautherized person is pronunautherized	methor control the national will in the particular the standard the particular to the particular to the solution of the solution to the solution of the soluti	



INON BALANCE IN CHOSSOVER

Runs 202 and 203

Iron in 45P	288 കൂ.	
Iron in chemicals added	2	
Total in		290 .m.
Iron in wastes (71-3P-3 & 4)	90	
Iron in 151R	. 160	** *** *** *** *** *** *** *** *** ***
Total out		250
bnaccounted for loss thru the	two crossover by-prod. pptns	-
Iron in 151R	160	
Iron in chemicals added	1.	
Total in		161
iron in waste (153.)	517	
lron in slurry (2 D1-S)	11	
Total out	•	528
Unaccounted for gain thru Cell	- 5	367

This large gain is probably largely due to the corrosion of the un-heat-treated welds made in forming the bends in the line from the centrifuge to the catch tank. This line has been replaced by a cold bent line, thus eliminating all but the flange welds.

Iron in Di-S (2 ch.s.)	21 gm.	
Iron in kOH	6	
Iron in other chemicals	1	
Total in		28 gm.
Iron in wastes (D3C)	12	
(D3:J.)	1	
fron in cake soln.	20	•
Total out		33
unaccounted for ain thru la	F 3 cake soln.	5

The KOH- $K_2\text{CO}_3$ metathesis appears to aid in the removal of iron carried in from Cell 5. The quantity carried in from Cell 5 can be expected to decrease with the use of the new line in Cell 5. The iron in the KOH will be decreased to about 1/8 of the above value by the use of a purer material.



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